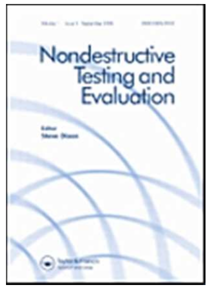

Nondestructive Examination of Recovery Stage during Annealing of a Cold-Rolled Low-Carbon Steel using Eddy Current Testing Technique

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Keywords:	Carbon steel, Eddy current, Nondestructive testing, Recovery, X-ray line broadening

Nondestructive Examination of Recovery Stage during Annealing of a Cold-Rolled Low-Carbon Steel using Eddy Current Testing Technique

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Abstract

The recovery process is usually investigated by conventional destructive tests that are expensive, time consuming and also cumbersome. In this study, an alternative non-destructive test technique (based on eddy current testing) is used to characterize recovery process during annealing of cold-rolled low-carbon steels. For assessing the reliability of eddy current results corresponding to different levels of recovery, X-ray line broadening analysis is also employed. It is shown that there is a strong relationship between eddy current outputs and the extent to which recovery occurs at different annealing temperatures. Accordingly, the non-destructive eddy current test technique represents the potential to be used as a reliable process for detection of the occurrence of recovery in the steel microstructure.

Keywords: Carbon steel; Eddy current; Nondestructive testing; Recovery; X-ray line broadening.

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1. Introduction

Movement and arrangement of dislocations directly affect plastic behavior of metals. At lower temperatures, deformation increases the dislocation density which is the main reason for strain hardening. Dislocations annihilation and rearrangement of them into energetically preferable configurations, as the main mechanisms for recovery, occur during low temperature annealing heat treatments performed in order to soften cold-deformed materials and produce a more desirable texture, to improve their formability [1, 2]. Annihilation of dislocations take place for dislocations with opposite signs and the other dislocations with equal signs start to arrange into lower energy arrangement and making small angle grain boundaries. Also, formed vacancies during forming process contribute to climbing of edge dislocations in the rearrangement procedure.

These modifications also result in the restoration of physical properties, i.e. magnetic and electrical properties of the steel [3]. Thus, it is of great practical importance to know whether recovery has happened during the annealing heat treatment process, whilst the ability to estimate the extent to which recovery takes place has potential application for industrial process optimization or as a laboratory tool. Transmission electron microscopy (TEM) can be successfully implemented to study the microstructure of metallic materials during recovery through observation of their dislocation structure formed as a result of deformation, however, disadvantages include the difficult and time-consuming sample preparation, particularly for a complete examination of the microstructure [4]. Other experimental techniques for the evaluation of changes in microstructures as a result of recovery include coercivity [2] and resistivity [5] measurements, tensile tests [6], X-ray line broadening [7] and peak resolution [8] techniques, ultrasonic testing [9], thermo-electrical power measurements [10], and microhardness evaluation [11]. These are less direct techniques when compared with TEM examination, but have the advantage of being applicable to bulk materials. However, these experimental techniques have at least one of problems such as having difficulties in carrying out of tests (thermo-electrical power measurements), relatively costly and time consuming in obtaining valid responses (X-ray line broadening and peak resolution techniques as well as tensile testing), studying only a small area (microhardness evaluation) and need for careful surface preparation (microhardness, coercivity and resistivity measurements) and therefore, they cannot be easily used on large commercial products. Also, some of these methods can't be used in some applications due to the destructive nature (tensile testing and X-ray techniques).

From an industrial point of view, application of nondestructive testing methods facilitates detection of microstructural changes in heat treated parts without suffering from disadvantages of conventional destructive tests. Using process integrated nondestructive tests proposes one of the possible way for materials characterization associated with saving energy and time along with providing 100% careful quality examination in the process of producing large numbers of products [12, 13].

Eddy current (EC), as a widely used test method for nondestructive examination of materials, is one of the applicable techniques for evaluation of the microstructural changes during the recovery stage of an annealing heat treatment cycle. EC technique is a magnetic nondestructive test method that can be used for accurate characterization of the microstructure of cold-worked steels during low temperature annealing treatments mainly due to its sensitivity to changes in the materials at the microscopic levels. The EC method works based on Faraday's law; placing a test sample in a coil with an induced alternating electromagnetic field results in the formation of ECs. These ECs produce a secondary electromagnetic field interacting with the primary electromagnetic field of the coil and hence, they cause measurable changes in the electromagnetic field produced by the coil [14-16].

Accordingly, it is expected that changes in the microstructure affect the eddy currents produced and they can be detected by calibrating the EC outputs [17].

EC test methods have been used for assessment of microstructural changes in steel, for example, phase changes [17-19], quality control of heat treated parts based on their grain size [14], evaluation of decarburizing depth [20], monitoring precipitation of intermetallic phases during aging treatments [21], microstructural changes detection during different steps of tempering heat treatment in AISI D2 tool steel [22], as well as evaluation of the degree of anisotropy of the microstructures [23].

In this research work, attempts have been made to evaluate changes in the microstructure during the recovery process in a cold-rolled low-carbon steel using EC nondestructive test technique. From the above list of available techniques for recovery investigations, only ultrasonic testing is comparable in simplicity and benefits to this technique. Thanks to the sensitivity of the EC method which has not yet been used for recovery study, useful method can be established for detection of changes during annealing of cold-rolled samples. Annealing the cold-rolled steel causes some changes in the density of dislocations and their spatial distribution in the microstructure, which are known to affect the magnetic properties of steel [1, 2], and therefore, EC outputs are expected to be affected by this process. X-ray line broadening testing was also used for assessing the annealed microstructures with the final goal of calibrating the EC responses of the material.

2. Experimental procedures

The starting material was a cold-rolled low-carbon steel having a microstructure consisting of mostly ferrite phase with small amount of pearlite (Fig. 1-a). The steel was initially cold-rolled to a thickness of 0.55 mm through a total reduction of 75%. The chemical composition of the investigated steel is 0.06 wt.% C, 0.20 wt.% Mn, 0.01 wt.% Si, 0.04 wt.% Ni, 0.01 wt.% Cr and 0.05 wt.% Cu. For producing different level of recovery whilst avoiding recrystallization, prepared samples were isothermally annealed at temperatures of 200 °C, 300 °C, 400 °C, 500 °C, 520 °C and 550 °C for various soaking periods of 1, 2, 3 and 4 hours. After heat treatment, samples were taken for conventional metallographic observations and Vickers's hardness measurements. At least 15 hardness measurements were averaged for each heat treating condition. X-ray diffraction (XRD) patterns of selected samples (as-rolled and isothermally annealed within the temperature range of 200 to 520 ºC for 2h) were obtained using a PHILIPS X-ray diffractometer implementing CuK_a radiation. To perform the scan, angular range of 40–90° having the step size of 0.02° as well as duration of 0.5 sec counting at each step were implemented to achieve the peak profile of the (200) pole. For recovery studies, both (200) and (222) poles has been extensively taken by researchers due to the better consistency with the microstructural changes in the carbon steels [8, 24-26]. In the current study, both poles were used, but the (200) peak was very sensitive and more accurate than (222). Therefore, (200) crystallographic peak was implemented. All samples were polished before XRD examination. Analysis of the peak profile broadening on the fitted curves, i.e. the full width at half maximum (FWHM) of the XRD peaks, was carried out using PANalytical X'pert High Score software.

Non-destructive tests were performed using a multi-frequencies EC instrument [14, 17] at room temperature in which the lift-off distance was zero (the distance between the sample and the coil). The device applies AC currents covering a broad range of frequencies (0.5 Hz to 5 MHz) to a flat pancake coil. Pick-up coil (500 turns of 0.2 mm insulated copper wire) was wounded on a ferritic core and 180 turns of 0.3 mm insulated copper wire was wounded on a ferritic cylinder which was around a pick-up coil. It has to be added that the design of flat pancake coil was done with respect to the shape of samples which were sheets. As expressed in equation of electromagnetic skin depth (Eq. 1), penetration of eddy currents controls by frequency (*f*), permeability (μ) and conductivity (σ).

$$\delta_{(f,\mu,\sigma)} = \frac{1}{\sqrt{\pi f \mu \sigma}} \tag{1}$$

Therefore, one of the most important steps in using EC is frequency determination. Two methods has been used in literatures [14, 17, 27, 28] for this purpose which are using the Eq. 1 as well as applying regression analysis on EC outputs. Here for finding the best optimum operating frequency, initial measurements were made on the impedance outputs at different frequencies from 1 to 25 kHz (in 0.5 kHz steps) and the best optimum operating frequency was determined by regression analysis [29] in which the optimum frequency was chosen while the best relationship between the EC output and materials properties obtained. The optimum operating frequency was determined in this way to be 5 kHz, which corresponded to the highest obtained correlation coefficient value.

3. Results and discussion

Figure 1 shows the microstructures of the deformed and annealed samples at different annealing temperatures. Figure 1-a shows the elongated deformed grains in the rolling direction. At annealing temperatures below 520 ºC, the microstructures are not significantly different from that of the as-rolled specimen suggesting that almost no recrystallization has occurred. However, at the annealing temperature of 520 ºC (Fig. 1-d), new recrystallized grains have nucleated, mostly on the grain boundaries of the initial deformed elongated grains. In the following (Fig. 1-e), recrystallization is almost finished (98% new recrystallized grains) for the annealing temperature of 575 ºC at the same soaking time. As can be clearly observed in Figures 1-b and 1-c, optical microscopic examination is not able to detect microstructural changes during recovery due to the occurrence of these changes being at the atomic (dislocation) scale. To assess the changes due to recovery, the FWHM XRD data for the as-rolled sample and the specimens annealed for 2 hours at different temperatures along with their Vickers's hardness values are given in Fig. 2. X-ray line broadening characterization of recovery was chosen in the present study as an alternative test technique for comparison to the data obtained from the EC tests, mostly due to the fact that it provides statistically averaged information for bulk materials, compared to TEM imaging [30]. X-ray line broadening relates mainly to the level of non-uniform lattice strain in the material. Rodriguez Torres et al. [24], have shown that X-ray line broadening, via monitoring the evolution of the XRD line width, i.e. FWHM, is a suitable technique for studying both recovery and recrystallization phenomena in low-carbon steels.

In Fig. 2, the FWHM decreases continuously from the annealing temperature of 200 ºC to 500 ºC, whereas the hardness exhibits no significant change in this temperature range. These observations are in agreement with those reported in the literature with regard to the annealing of low-carbon steels [2, 24]. In the recovery region, where the hardness of the specimen remains almost unchanged, the decrease in FWHM values exhibit the progress of recovery via the dislocations annihilation and the rearrangement of others into lower energy levels [2, 8]. The strain induced X-ray line broadening arises from crystal imperfections and distortion which are related to the changes produced during cold work [25]. From the dislocation models [31], it is known that the lattice curvature around dislocations results in incoherent X-ray reflection over distances much greater than the dislocation separation. Early studies [31], have shown that cold working can increase the dislocation density up to approximately 10^{12} cm^{-2}. Therefore, the greater the density of dislocations, the larger would be the general broadening of the X-ray line. Recovery reduces the overall strain energy of the lattice (through lowering the stored energy of the lattice) and therefore (as observed in Fig. 2), the FWHM decreases with increasing annealing temperature.

The increasingly decrease in FWHM with the increase in annealing temperature continues up to 520 ºC which is the temperature for formation of some new dislocation-free recrystallized grains in the steel microstructure (see Fig. 1-d). At this point, a significant drop takes place for hardness results which can be attributed to the onset of recrystallization which is consistent with other results. Finally, last point represents hardness for the sample annealed at 575 ºC for 2h which is almost fully recrystallized.

According to the experimental results obtained in this study, hardness measurements and metallographic observations showed almost no sensitivity to the recovery processes, however, XRD investigations indicate that recovery has occurred progressively with annealing temperature. Hence, XRD results were used to check the validity of the EC non-destructive results.

EC testing was carried out to determine if it could characterize the amount of recovery in the steel samples as well as the onset of recrystallization. The EC outputs, in terms of normalized impedance vs. different annealing treatments, are shown in Fig. 3 for the different annealing temperatures. The normalized impedance value is obtained by dividing the measured impedance (Z) by the impedance of the empty coil (Z_0) [14]. Observing Fig. 3, the normalized impedance values increase with an increase in soaking times at a given annealing temperature. Also, impedance values increase for a specific time with an increase in the annealing temperatures. For categories of 1h and 2h it happens in the range of below 500 ºC and for 3h and 4h it can be seen below 400 ºC. As mentioned already, these changes in impedance correspond to the occurrence of recovery.

The variation of normalized impedance with annealing temperature in Fig. 3 depends on the magnitude of the ECs, where the induced ECs are functions of physical parameters such as edge effect, lift-off and skin effects as well as the electromagnetic parameters including the operating frequency, magnetic permeability and electrical conductivity of the material [17, 32]. In this study, the conductivity and permeability (i.e. materials parameters) are the only effective parameters that affect the ECs, since the physical parameters and test frequency are fixed. In particular, the magnetic permeability shows a dominant effect in ferromagnetic alloys [14]. Here, any difference in the magnetic permeability is assumed to be due to the microstructural changes.

As mentioned already, at the annealing temperatures where only recovery takes place (for instance, in the range of 200 ºC-500 ºC for 2 h) the normalized impedance values increase progressively compared to the as-rolled sample value. Increasing the annealing temperature (i.e. increasing the degree of recovery corresponding to a reduction in dislocation density and lower-energy arrangement of dislocations) results in a lower resistance of the material to the magnetic field. This, in turn, makes the amount of magnetic permeability (μ) greater. Considering Eq. (2) [29], this increase in the permeability increases the self-induction coefficient, L, i.e.:

$$L = \mu N^2 A / l \tag{2}$$

where, N, A and l are the number of coil turns, the area of cross section and length of the coil, respectively.

Based on Eq. (3) [29], increasing the self-induction coefficient gives rise to the increase in the induction resistance (X_L), and this increases the impedance, Z, (Eq. (4)). It should be borne in mind that in Eq. (4), the effect of permeability in ferromagnetic alloys such as low-carbon steels is stronger than that of the resistance, R [14].

$$X_L = 2\pi f L \tag{3}$$

$$Z = \sqrt{X_L^2 + R^2} \tag{4}$$

The progress of recovery depicted in Fig. 3 indicates that with the increase in annealing temperature and also soaking time, the cold-rolled samples reach a higher level of recovery due to the increased reduction of dislocation density. These EC results indicate that more complete recovery occurs at a higher annealing temperature or for a longer soaking time.

Another important set of experimental results obtained from the eddy current examination is the entirely different behavior of the investigated steel at higher annealing temperatures. With the increase of annealing temperature for each soaking time, normalized impedance eventually decreases drastically owing to the onset of recrystallization (after 400°C for 3 and 4 hours, after 500°C for 2 hours and after 520°C for 1 hour soaking). The volume fractions of new recrystallized grains formed in the steel microstructures at these annealing conditions were measured to be 7, 17, 14 and 10%, respectively. Therefore, one may speculate that this significant drop in the normalized impedance is about the appearing of very small strain free grains during recrystallization at the mentioned annealing temperatures and times. For samples annealed for 2h, drastic decrease continues by sample annealed at 575 °C which corresponds to the almost fully recrystallization. EC output drop is a conclusive proof which shows that the strain free grains are appearing at a suitable rate, so grain refinement (increases magnetic permeability) has a dominant effect as compared to dislocation density reduction (decreases magnetic permeability), which is consistent with the results of other authors [2]. In addition, this series of experimental results are consistent with those obtained by Gurruchaga et al., [33], for the case of annealing a cold-rolled high carbon steel at the temperatures in the range of 300 to 575 °C for different periods of time by means of magnetic Barkhausen noise non-destructive method. They have shown that the amplitude of the magnetic Barkhausen noise envelopes increased progressively with annealing time at the lower annealing temperatures (300-500 °C). At the annealing temperature of 575 °C where recrystallization was shown to start after 100 seconds, on the other hand, a significant drop was seen in the height of the Barkhausen peaks for the soaking times longer than 100 seconds at this temperature.

The Experimental results in the present investigation obtained in different annealing conditions exhibit similar trend. As mentioned before, after annealing for one hour and 4 hours at 550 and 500 °C, respectively, corresponding the formation of 10 and 17% recrystallized grains, the normalized impedances have considerably decreased suggesting that the grain boundaries of both recovered elongated grains and relatively small recrystallized grains of submicron size are predominant microstructural parameters which significantly affect the electromagnetic responses of the steel.

In our previous work [14], where the effect of grain size on EC output was investigated, the reduction in grain size (increasing the grain boundary area density) resulted in more resistance to the passage of the magnetic field which, in turn, caused a reduction of the magnetic permeability. This reduction in permeability led to a reduction in the EC output (see Eqs. (2) - (4)). Therefore, the onset of recrystallization and the corresponding changes in microstructure is responsible for the observation of impedance drop in Fig. 3.

Although the application of eddy current electromagnetic induction for the evaluation of microstructural changes during recrystallization in steels needs separate detailed investigations in order to find the main controlling mechanism(s), one may consider eddy current technique a powerful and reliable test, based the results of the present study, to evaluate nondestructively the beginning of recrystallization after the recovery stage.

4. Conclusions

To summarize, recovery progress during annealing of a cold-rolled low-carbon steel was successfully investigated using X-ray line broadening and EC techniques. On one hand, FWHM, as an indicative of measured changes by XRD, decreased continuously in the recovery region due to the elimination of excess point defects as well as the dislocations annihilation and rearrangement of them into lower energy configurations. It was found that hardness measurements and metallographic observations were not sensitive to recovery progress. On the other hand, EC was found to be a suitable alternative technique for studying nondestructively the progress of recovery. It was showed that EC is very sensitive to microstructural changes during recovery which were not detected by metallographic observations. In addition, with reference to the contradictory effects of recovery and recrystallization on the EC outputs, one can use EC method to differentiate between the recovery and recrystallization processes during an annealing heat treatment cycle and therefore, to detect experimentally the onset of recrystallization of deformed steel microstructures. EC outputs increased in recovery progress, but this trend was changed as a result of formation of new recrystallized grains in recrystallization process, so grain refinement has a dominant effect on EC outputs as compared to dislocation density reduction.

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Figures Captions:

Fig. 1. Optical micrographs showing the starting material (a) and test samples annealed for 2 h at different annealing temperatures of 300 °C (b), 500 °C (c), 520 °C (d) and 575 °C (e).

Fig. 2. Variations of the FWHM and Vickers's hardness number with the annealing temperature for samples annealed for 2 hours.

Fig. 3. The normalized impedance as a function of annealing temperature for different soaking times.

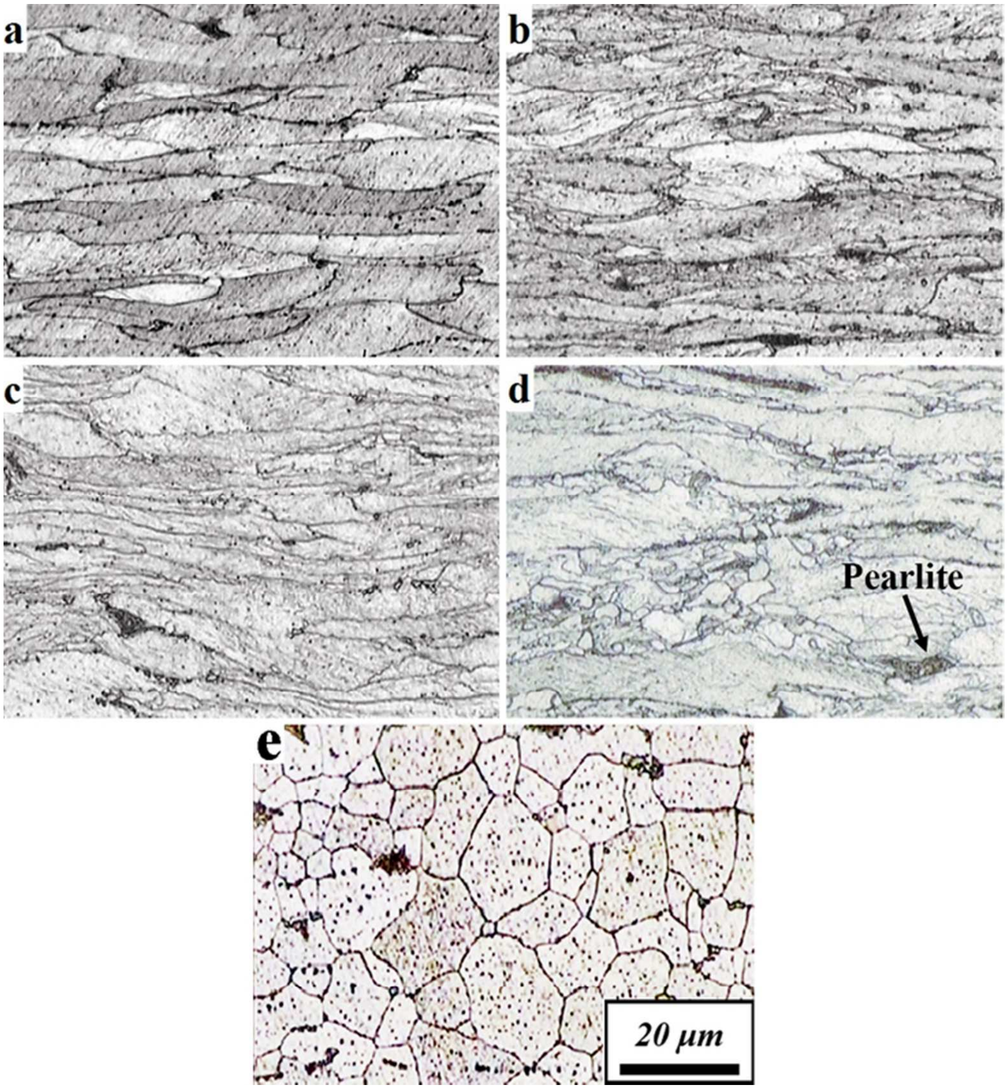

Fig. 1. Optical micrographs showing the starting material (a) and test samples annealed for 2 h at different annealing temperatures of 300 °C (b), 500 °C (c), 520 °C (d) and 575 °C (e).

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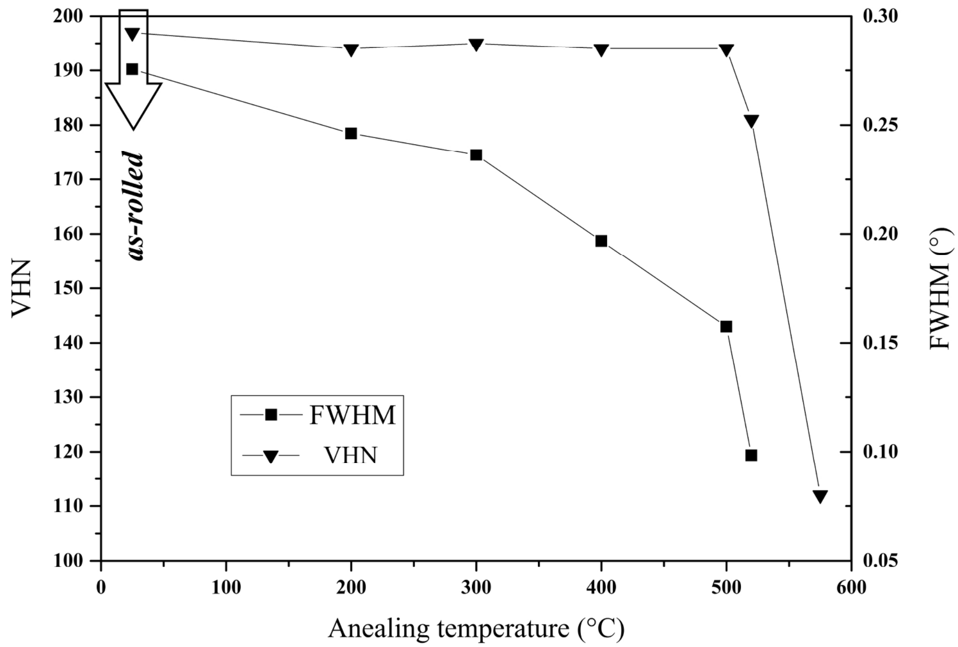

Fig. 2. Variations of the FWHM and Vickers's hardness number with the annealing temperature for samples annealed for 2 hours.

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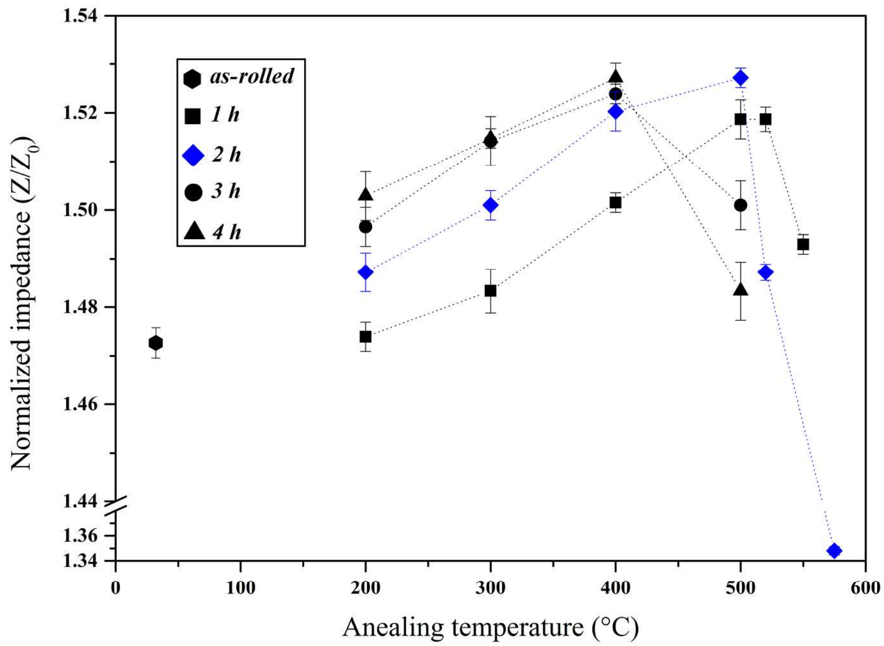

Fig. 3. The normalized impedance as a function of annealing temperature for different soaking times.

111x81mm (600 x 600 DPI)

Ms. Ref. No.: GNTE-2016-0106.R1

Title: Nondestructive Examination of Recovery Stage during Annealing of a Cold-Rolled Low-Carbon Steel using Eddy Current Testing Technique

Nondestructive Testing and Evaluation

Dear Prof. Edwards,

Receiving subtle reviewer's comments, we did the revision according to the optional comments. The detailed corrections as well as the authors' answers to the reviewers are listed below.

We look forward to your positive response.

With the best regards and wishes,

Sadegh Ghanei
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27 October 2017

Reviewer's comments:

Referee: 1

Comments to the Author

Dear Authors,

The manuscript is well written and targets important subject of non-destructive testing of low carbon steel using eddy currents and evaluate the material's recovery state. The conclusions are sound and provide a basis for furthering, validation and generalization of the proposed method in ferrous materials.

***Response**: We appreciate the effort of the reviewer in deep review of our manuscript and making this paper perfect with the valuable comments.*

It would be optionally desirable if the Authors could mention that in Results and Discussion Section, 7th page, 41 line that the first recrystallization state is finished.

***Response**: Recrystallization is identified with two different stages of nucleation and growth of the new strain-free grains (1). As can be seen in Fig 1.d, a few strain-free grains nucleated which represents the onset of recrystallization. Therefore, it is not desirable to tell that whole first stage (nucleation) was finished because it confuses the reader. Thus, we did not apply this optional point in the text.*

Additionally the XRD broadening method (the choice of (200) crystallographic ferrite peak) could be commented in more detail referenced through eventual convenient previous publication(s).

The manuscript can be accepted for publication after these minor optional revisions.

***Response**: For recovery studies, both (200) and (222) poles has been extensively taken by researchers due to the better consistency with the microstructural changes in the carbon steels [8, 24-26]. In our study, we studied both poles but, the (200) peak was more accurate than (222). Therefore, we chose (200) crystallographic peak. According to this optional comment, the relevant sentences and corresponding references were added to the manuscript in page 5 and highlighted in green colour.*

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1. Campbell FC. Elements of Metallurgy and Engineering Alloys: ASM International; 2008.